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13. ABSTRACT (Maximum 200 words)

Our primary goal is the measurement of the third order elastic constants of solids using resonance ultrasound spectroscopy (RUS). Progress on the construction of a new apparatus by which RUS measurements are made at elevated pressure is described. We chose materials which provide optimal values of αK_T for our initial set of measurements, and describe the relevant thermodynamic relations. Initial pressure runs with helium and argon pressurizing mediums for fused silica and KCl have been made, but their interpretation requires further analysis of the spectral data. Considerations regarding the measurement of third order elastic constants that are not obtained from purely hydrostatic stresses are presented. Completed RUS projects regarding the specimen size effects on measured elastic moduli, the elastic properties of $Fe_{0.943}O$ and the elastic/viscoelastic properties of iron are summarized.

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Measurement of the Pressure Derivatives of Elastic Constants Using Resonance Ultrasound Spectroscopy

Principal Investigator: Orson L. Anderson

Annual Summary Report for May 30, 1994–May 30, 1995

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Experiment

A. Description of Project and Approaches

Our primary experimental goal is to measure the third order elastic constants of several materials using resonant ultrasound spectroscopy (RUS). RUS experiments are currently underway at elevated hydrostatic (gas) pressure for fused silica and KCl. These materials were chosen for initial test specimens because their combined values of thermal expansion and bulk modulus indicate that they should give relatively large frequency changes at elevated pressure compared to temperature induced frequency changes. A critical experimental consideration is the extent to which frequency shifts due to small variations in temperature will mask pressure effects on the modal frequency. The ratio $(\partial f_{\alpha\beta}/\partial P)_T / (\partial f_{\alpha\beta}/\partial T)_P$, where $f_{\alpha\beta}$ are the modal frequencies, is essentially controlled by $(\alpha K_T)^{-1}$. Thus we selected materials with low values of αK_T for our initial set of experiments. Furthermore, in order to make precise temperature corrections, we made careful measurements of the frequency shifts due to temperature alone on the specimens we are now placing under pressure.

In addition to concerns about corrections for temperature, we must consider effects due to the pressurizing gas medium. As pressure increases there is an increased coupling between the specimen and the surrounding gas which cause a decrease in amplitude of the modal frequencies and introduces an effective mass loading effect on the specimen. The problem with amplitude limits the high pressure range of our experiments, but this can be partially compensated for by making extremely accurate pressure and temperature measurements over the allowable pressure range. The mass loading of the pressurized gas on the specimen is more problematic. One way we are addressing this problem is by making measurements with pressurizing gases of varying molecular masses. We are also in consultation with Moises Levy (University of Wisconsin, Milwaukee) regarding theoretical means by which mass loading effects can be decoupled from the frequency shifts due to pressure alone.

Our recent measurements using helium gas up to 100 bar on fused silica show a systematic decrease in resonant frequencies with increasing pressure. This type of shift is expected since the bulk and shear moduli for fused silica decrease (increase) with increasing pressure (temperature). We have obtained further data using an argon pressurizing medium, but have not yet analyzed these results. Pressure runs have also been performed with helium gas on KCl to 150 bar, but these data are also not yet analyzed. We expect to report our initial results at the Second Annual Meeting of the Consortium on Resonant Ultrasound Spectroscopy, August 24-26, 1995.

B. Other Accomplishments Completed During Past Year

During the past year we completed the construction and testing of our new pressure generating and measuring equipment. Specific objectives that were met are as follows:

1. Finished mounting and assembling the dead weight tester.
2. Completed testing of high pressure vessel/jacket pressuring system.

3. Inserted a differential pressure cell in line with the dead weight tester and pressure vessel.

4. Finished constructing and testing sample holding apparatus for insertion into the high pressure chamber.

5. Completed tests for temperature gradients and temperature equilibration time in the pressure cell at elevated pressure.

Further RUS experimental projects that were completed during May 30, 1994, to May 30, 1995, include the following:

1. Specimen size effects on the measured elastic properties with RUS.

   Published data on Fe_xO reveal that the value for the bulk modulus determined from RUS is anomalously high. Since the size of the specimen in the RUS experiment was small (~ 1 mm^3), some investigators have suggested there is a systematic bias in RUS values as the specimen size becomes small. We performed measurements on fused silica to measure the extent to which specimen size affects the measured elastic properties. We used five specimen volumes ranging from 44.5 mm^3 to 1.5 mm^3. No systematic effects from specimen size are found. Results from data reduced at zero mass holding force from both the highest and lowest volumes used agree within the experimental errors. We also find agreement between the zero-mass holding force data and data reduced from a holding force equivalent to the weight of two grams. These results indicate that there is no measurable size effect in RUS experiments down to the smallest volume we measured, and provide confidence that specimen size consideration is not relevant to future pressure experiments.

2. Measurements of the elastic moduli for $Fe_{0.943}O$.

   We also addressed the question of the elastic properties $Fe_{0.943}O$ by RUS measurements on a specimen cut from the same boule as a specimen for which ultrasonic pulse-superposition measurements were obtained. We find general agreement with the pulse-superposition data. However, the RUS data reduction for $Fe_{0.943}O$ presents peculiar difficulties in that the computed values for the compressional moduli are very sensitive to changes in measured frequencies. We are currently investigating this situation.

   We investigated the softening of the C_{44} modulus of Fe_xO by performing measurements up to 500 K on $Fe_{0.943}O$. This softening originates with the magnetic transition near 180 K. Previous studies have documented the effects of this magnetic transition in the anomalous behavior of the C_{44} modulus (C_{44} increases [decreases] with increasing temperature [pressure]) from below 180 K to room temperature. No published data are available for the elastic properties of Fe_xO above room temperature. We find that the mode softening extends to at least 500 K, the upper limit of our temperature data. Future plans include the investigation of the pressure effects on the C_{44} modulus of Fe_xO using RUS.

3. The elastic and viscoelastic properties of α-iron.

   The analysis of the RUS data on α-iron to determine the extent to which viscoelastic effects are present at high temperature was completed. These data are relevant to attempts at interpreting the low shear wave velocities relative to the compressional wave velocities within Earth's inner core. Our results provide evidence that viscoelastic effects on shear wave velocities (or on the rigidity modulus, mu) are present, but are considerably less, than previous studies have indicated. Furthermore, when our results are compared with other ultrasonic and torsional data, we find no evidence of dispersion in high temperature measurements of μ at frequencies of 3 Hz or greater. Other data (torsional) show a considerable degree

of dispersion in the 0.001–1 Hz range at high temperature. Our results and analysis: (a) better quantify the extent to which dispersion in measured values for μ must be accounted for when comparing low frequency data (<1 Hz) and ultrasonic frequencies; (b) indicate that the range of seismic frequencies (0.1–10 Hz) may contain frequencies where dispersion in μ is both present and otherwise; and (c) provide support for interpreting the relatively low shear wave velocities in Earth's inner core in terms of viscoelastic phenomenon in the solid phase of iron.

Theory

Theoretical work has proceeded along three lines: 1) to examine the calculation of the cross derivative $\partial^2 K_T/\partial T \partial P$ arising from measurements of frequency versus P along isotherms; 2) to examine whether important thermal properties, like the entropy, can be determined from the results of our experiments; 3) to design experiments that measure the third order elastic constants which are independent of pressure.

On Point 1, we found that the measured frequencies are very sensitive to temperature because are our experiments are limited to a small pressure range. We must therefore evaluate $\partial^2 K_T/\partial T \partial P$ in order to construct an accurate data set from measured RUS isotherms. It turns out that the crucial parameter to evaluate $\partial^2 K_T/\partial T \partial P$ is related to specific heat, $(\partial \ln C_P/\partial \ln V)_T$. This comes from a thermodynamic identity relating $\partial^2 K_T/\partial T \partial P$ to thermoelastic constants including $\delta_T = -(1/\alpha K_T)(\partial K_T/\partial T)_P$. The trouble arises from the evaluation of $\partial \ln \delta_T/\partial V$. This parameter, if evaluated from experiments, needs the value of $(\partial \ln C_V/\partial \ln V)_T$. Up to now, we have assumed that $(\partial \ln C_P/\partial \ln V)_T$ is independent of V in our measurement range; but now, we are not sure, so we are now in the middle of an investigation to find the extent to which $(\partial \ln C_P/\partial \ln V)_T$ is independent of V, and the temperature range over which this is the case. In this investigation we use data of well-known solids, MgO and NaCl. This will lead to at least one paper.

On Point 2: very accurate isotherms of $C_{ij}(P)$ and very accurate isobars of $C_{ij}(T)$ can be determined from RUS. With the help of the thermal expansivity, isotherms of $C_{ij}(V)$ and isochores of $C_{ij}(T)$ can also be determined. This means that the product, αK_T, where K_T is the isothermal bulk modulus, can be determined as a function of V and T. The knowledge of αK_T versus V leads to entropy because of Maxwell's equation,

$$\left(\frac{\partial S}{\partial V}\right)_T \equiv \left(\frac{\partial P}{\partial T}\right)_V \equiv \alpha K_T.$$

This allows us to find the entropy as a function of V along isotherms. Thus we determine entropy without the usual thermal input, measured specific heat or measured enthalpy. All the thermal information is contained within the variation of K_T with T and with $\alpha = (1/V)\,dV/dT$. Since C_V is (dS/dT), we can gain knowledge of C_V and thus C_P. Thus, we see that by adding thermal expansivity to our measurement capacity, we can find many thermal properties and solve the problem we discussed in Point 1. A paper on this technique was written and has been published in *J. Chem. Phys.*

On Point 3, we are contending with the problem that in the measurement of certain third order elastic constants, a constant shear strain must be imposed on the solid. A problem is that an imposed shear strain would most certainly inhibit resonant vibrations from occurring. So far, we are stymied on designing an experiment to solve this problem.